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Solubility of RDX, PETN, and Boric Acid in Methylene Chloride

by Rose Pesce-Rodriguez

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14. ABSTRACT
This report provides the results of a study to determine the solubility of RDX, pentaerythritol tetranitrate (PETN), and boric acid in methylene chloride. Prior to determining the solubility, I examined the samples using desorption-gas chromatography-mass spectrometry. I found that the RDX and PETN contained residual solvents, so I dried those samples before analyzing them to determine solubility. The solubility of RDX and PETN at room temperature in methylene chloride was successfully determined and found to be 2.9 mg/mL and 8.0 mg/mL, respectively. The solubility of boric acid in methylene chloride was determined to be <0.004 mg/mL.

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Contents

List of Figures iv

List of Tables iv

1. Background 1

2. Experimental 1

2.1 Samples 1

2.2 Desorption-Gas Chromatography-Mass Spectrometry 1

2.3 Solubility Determination 2

3. Results and Discussion 2

3.2 Residual Solvents 2

3.3 Solubility Determination 2

4. Conclusion 3

Appendix. Gas Chromatograms and Mass Spectra (Including Library Matches) from Analysis of RDX and PETN by D-GC-MS 5

Distribution List 8

List of Figures

Figure A-1. Total ion chromatogram resulting from D-GC-MS analysis of PETN (air peak is an experimental artifact.) 6

Figure A-2. Mass spectrum of 1.66-min peak from figure A-1 (water). 6

Figure A-3. Mass spectrum of 1.9-min peak from figure A-1 along with library match (acetone). 7

Figure A-4. Selected ion chromatograms resulting from D-GC-MS analysis of PETN. 7

Figure A-5. Total ion chromatogram resulting from D-GC-MS analysis of RDX (air peak is an experimental artifact.) 8

Figure A-6. Mass spectrum of 1.66-min peak from figure A-5 (water). 8

Figure A-7. Mass spectrum of 1.7-min peak from figure A-5 along with library match (isopropanol). 9

Figure A-8. Selected ion chromatograms resulting from D-GC-MS analysis of RDX. 9

List of Tables

Table 1. Solubility results. 2

1. Background

At the request of Dr. Thuvan Piehler, who was interested in preparing explosive samples containing RDX, pentaerythritol tetranitrate (PETN), and boric acid in a binder by means of a solvent process, I determined the solubility of those materials in methylene chloride using high performance liquid chromatography (HPLC). I also analyzed RDX and PETN samples to determine if they contained any residual solvent.

2. Experimental

2.1 Samples

All samples were provided by Dr. Thuvan Piehler (U.S. Army Research Laboratory). The RDX used was Class 5 RDX from Holston Army Ammunition Plant, Kingsport, TN (Lot no. HOL88M675-079; Stock no. NSN1376000074877). The PETN was superfine PETN from Ensign-Bickford Aerospace and Defense Company, Simsbury, CT. The boric acid was granular, ACS Reagent Grade from Mallinckrodt Laboratory Chemicals, Phillipsburg, NJ.

2.2 Desorption-Gas Chromatography-Mass Spectrometry

I performed the analysis of residual solvent in RDX, PETN, and boric acid by means of desorption-gas chromatography-mass spectroscopy (D-GC-MS). Desorption was achieved via a CDS Model 2000 Pyroprobe®[*] (coil type) connected through a heated interface chamber to the splitless injector of an Agilent GC-MS system (model 6890N GC and model 5973N MSD). The GC column used was a HP-5 capillary column (0.25 mm × 30 m, 0.25 μm film). The injector temperature was 200 °C. The GC oven temperature program was as follows: 50 °C isothermal for 1 min, 50–250 °C at 40 °C/min, and 250 °C isothermal for 1 min. Samples were held within the coil of the Pyroprobe by first placing them in a quartz tube containing a small plug of glass wool, and then inserting the entire tube into the coil. For desorption analyses, the Pyroprobe was used either only to hold the sample in the interface (and not heat it further) or was programmed to give a 20-s desorption pulse at 175 °C (heating rate: 1000 °C/s). In all cases, the Pyroprobe interface temperature was 175 °C. Methylene chloride was analyzed by direct injection through a septum into the pyrolyzer interface (Pyroprobe removed).

[*]Pyroprobe is a registered trademark of Cds Analytical, Inc., Chemical Data Systems, Inc.

2.3 Solubility Determination

Standard solutions were prepared by adding pre-weighed pure compounds to known volumes of distilled water. I determined the absorbance maximum for each compound by means of an HPLC with a diode array detector. Solubility samples were prepared by adding excess pure, compound to distilled water. To ensure that the solution was saturated with the compound, I ensured that solid material was always present in the solution. Prior to analysis, the solution was centrifuged (3000 rpm, 20 min at 24 °C). Samples were analyzed at the predetermined λ_{max} and, if necessary, diluted and re-analyzed. Concentration was determined in mg/mL. For HPLC analysis, an Agilent Technologies (Santa Clara CA) 1200 Series HPLC was used. The solvent system consisted of 50% acetonitrile and 50% water. The flow rate was 1 mL/min, and the column was an Agilent Technologies Pinnacle II C18 5-µm (250 × 4.6 mm). The column was kept at a constant temperature of 24 °C. An injection volume of 10 µL was used.

3. Results and Discussion

3.2 Residual Solvents

I observed using D-GC-MS that both the RDX and PETN contained residual solvents. RDX contained water and isopropanol; and PETN contained water, isopropanol, and acetone. Both materials were dried before analysis. GC and MS library matches are given in the appendix. Based on analysis using D-GC-MS, the methylene chloride and boric acid did not show impurities of any sort.

3.3 Solubility Determination

Following the residual solvent analysis, I determined the solubility of RDX, PETN, and boric acid in methylene chloride. Table 1 shows the results. The value given for boric acid is an upper limit, as it was not possible to determine an actual value because the amount is so low. The value 0.004 mg/mL was the lowest concentration solution prepared at which the solute could be accurately weighed and visually observed in the methylene chloride.

Table 1. Solubility results.

Compound	**Solubility in Methylene Chloride (mg/mL)**
RDX	2.9
PETN	8.0
Boric acid	<0.004

4. Conclusion

The solubility of RDX and PETN at room temperature in methylene chloride was successfully determined and found to be 2.9 mg/mL and 8.0 mg/mL, respectively. The solubility of boric acid in methylene chloride was determined to be <0.004 mg/mL.

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Appendix. Gas Chromatograms and Mass Spectra (Including Library Matches) from Analysis of RDX and PETN by D-GC-MS

Figures A-1 through A-8 provide the GCs and MS (including library matches) from the analysis of RDX and PETN using D-GC-MS.

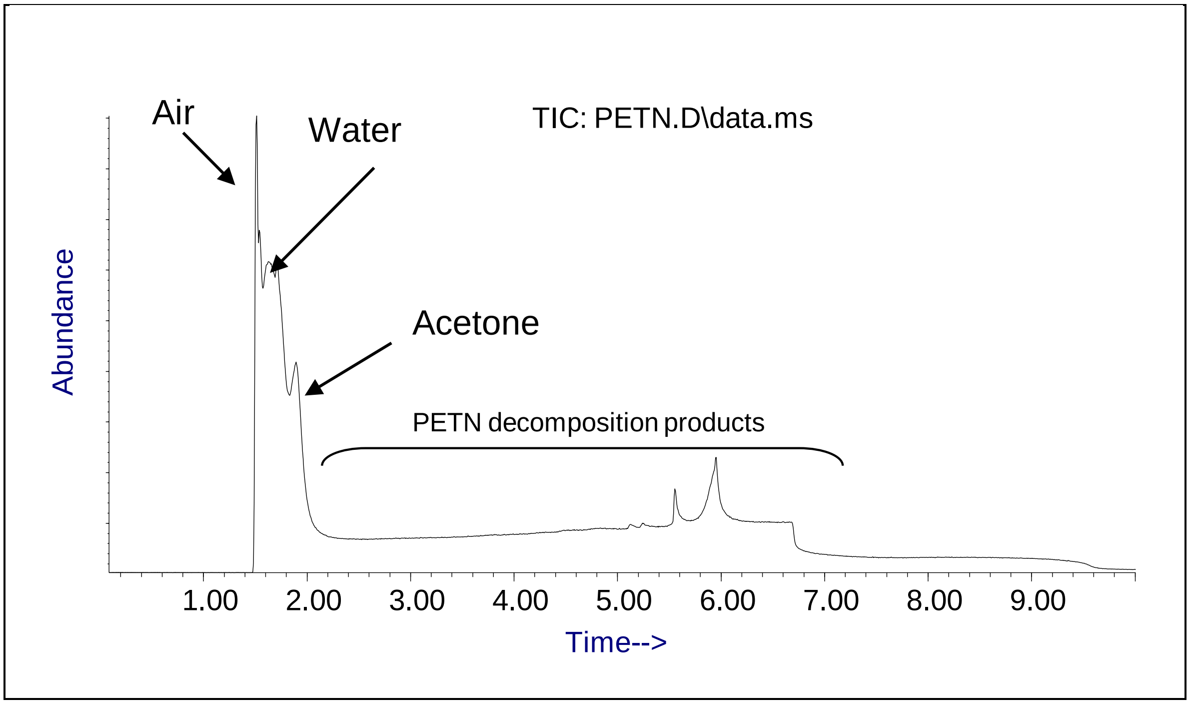

Figure A-1. Total ion chromatogram resulting from D-GC-MS analysis of PETN (air peak is an experimental artifact.)

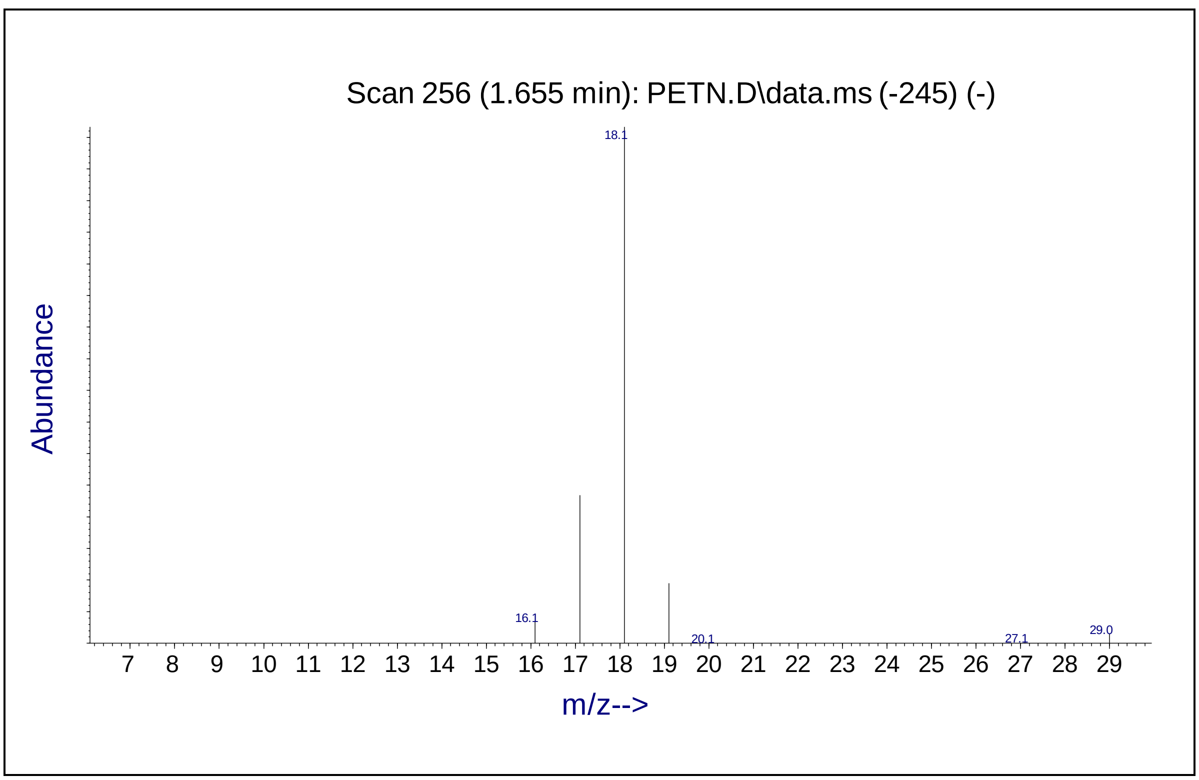

Figure A-2. Mass spectrum of 1.66-min peak from figure A-1 (water).

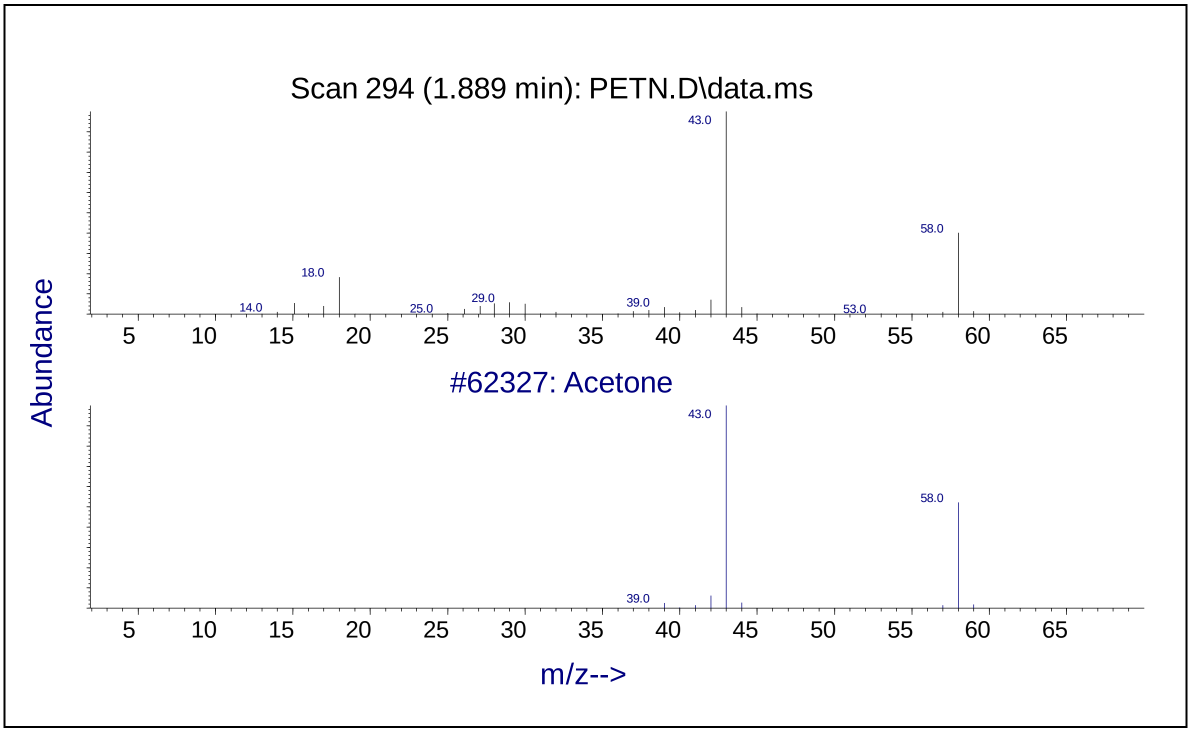

Figure A-3. Mass spectrum of 1.9-min peak from figure A-1 along with library match (acetone).

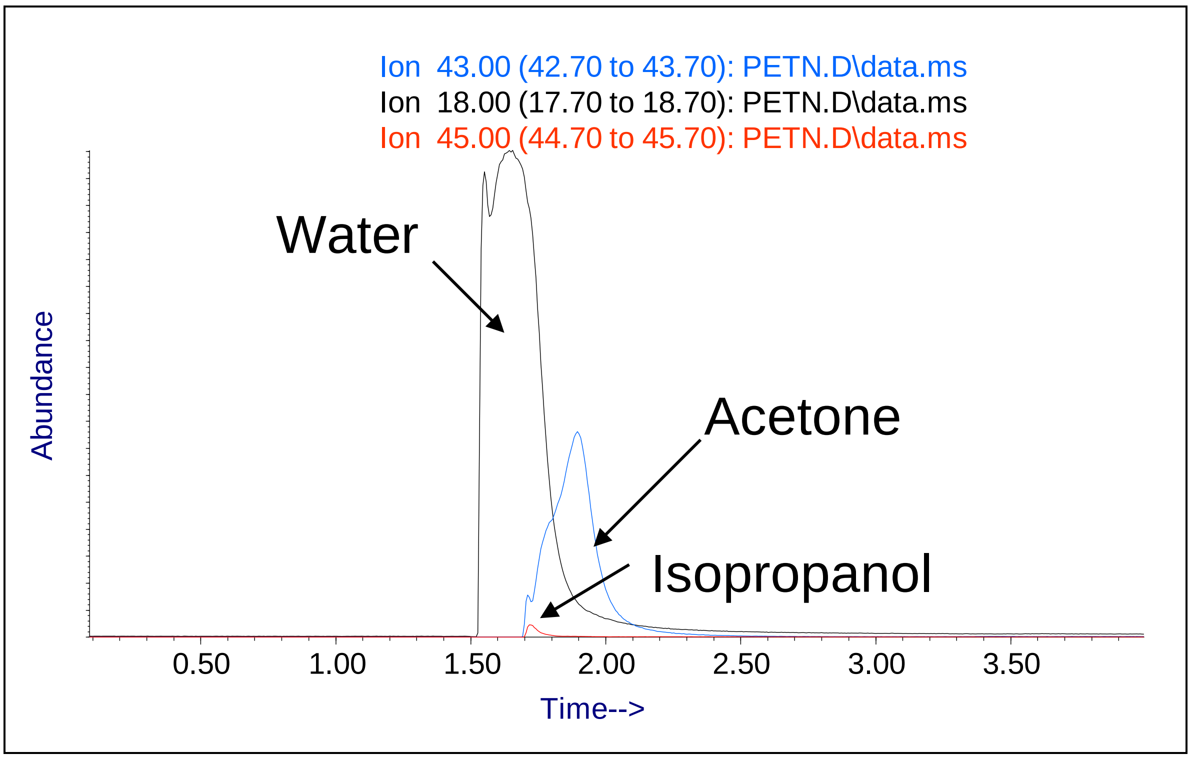

Figure A-4. Selected ion chromatograms resulting from D-GC-MS analysis of PETN.

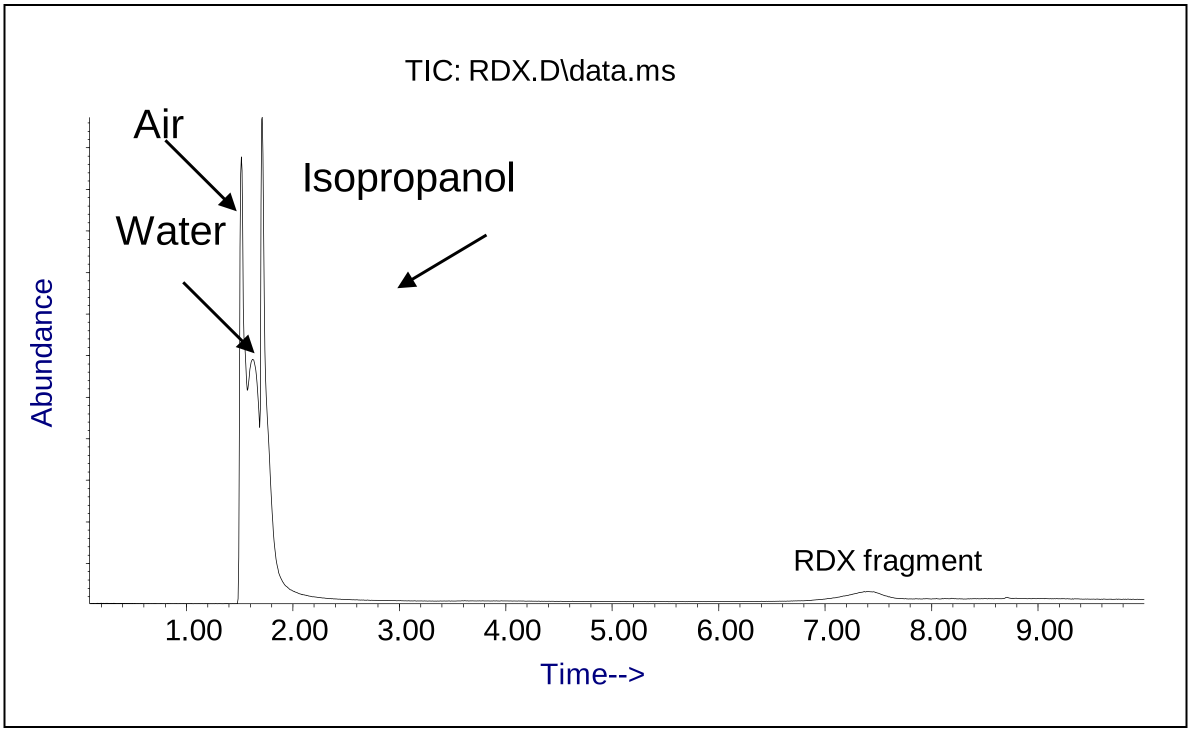

Figure A-5. Total ion chromatogram resulting from D-GC-MS analysis of RDX (air peak is an experimental artifact.)

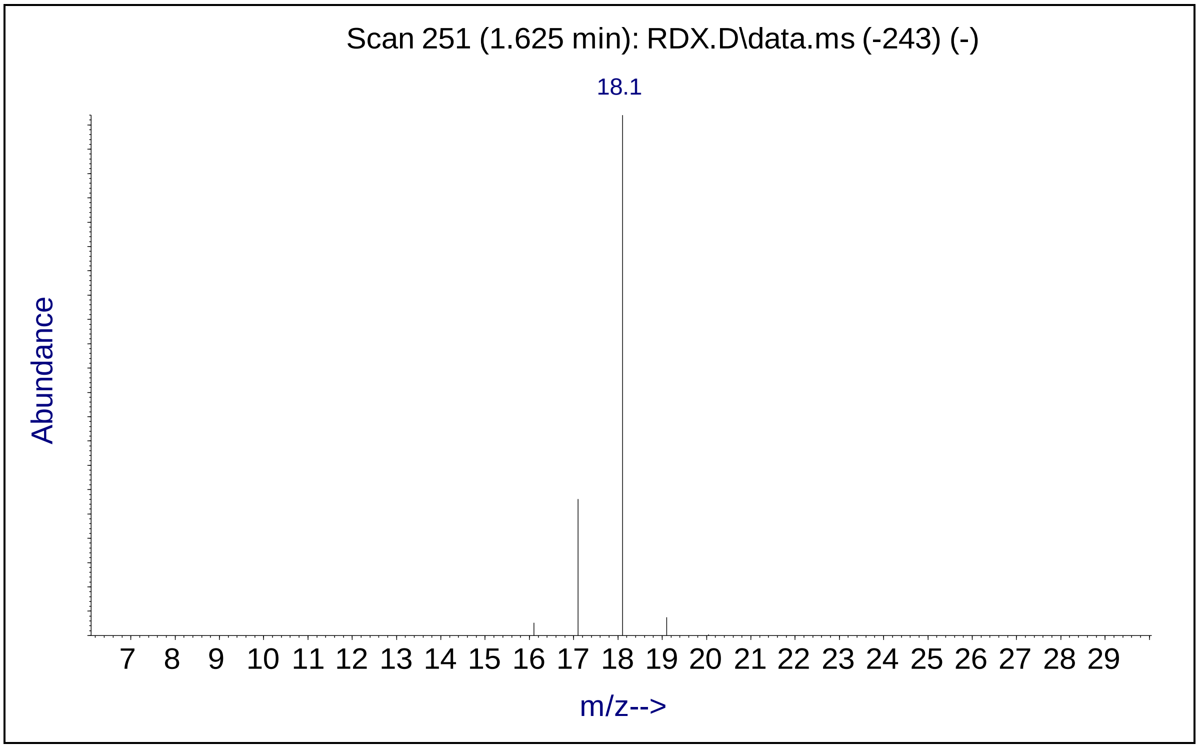

Figure A-6. Mass spectrum of 1.66-min peak from figure A-5 (water).

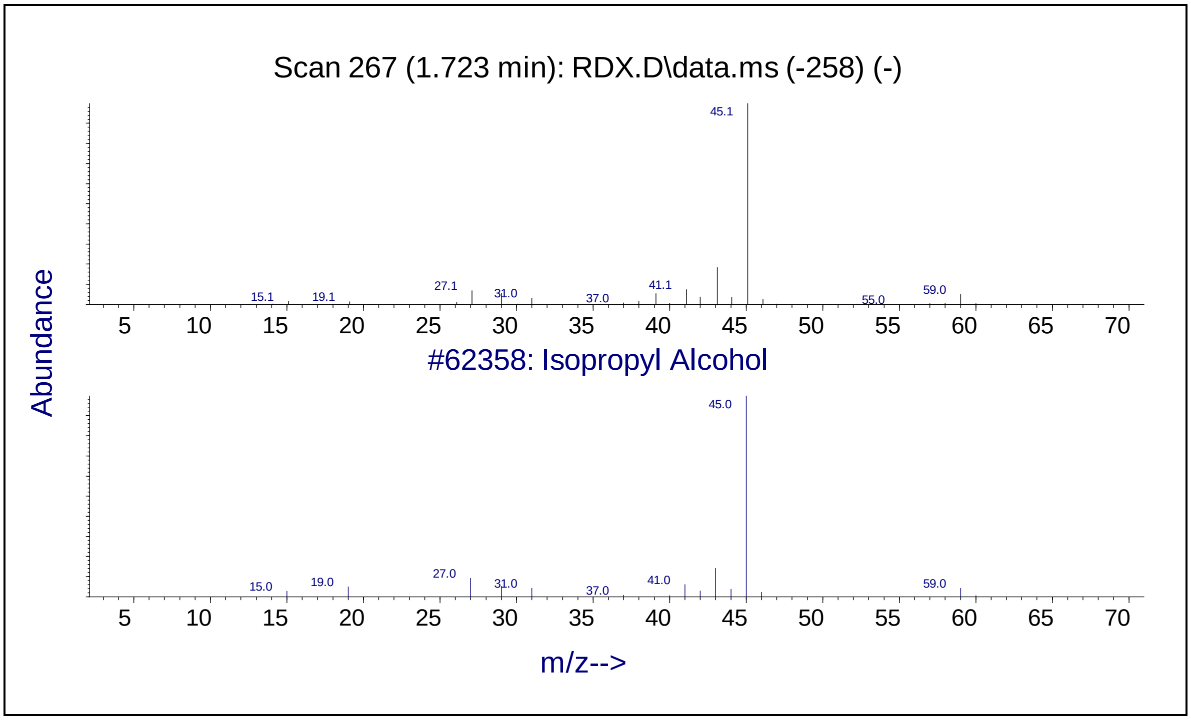

Figure A-7. Mass spectrum of 1.7-min peak from figure A-5 along with library match (isopropanol).

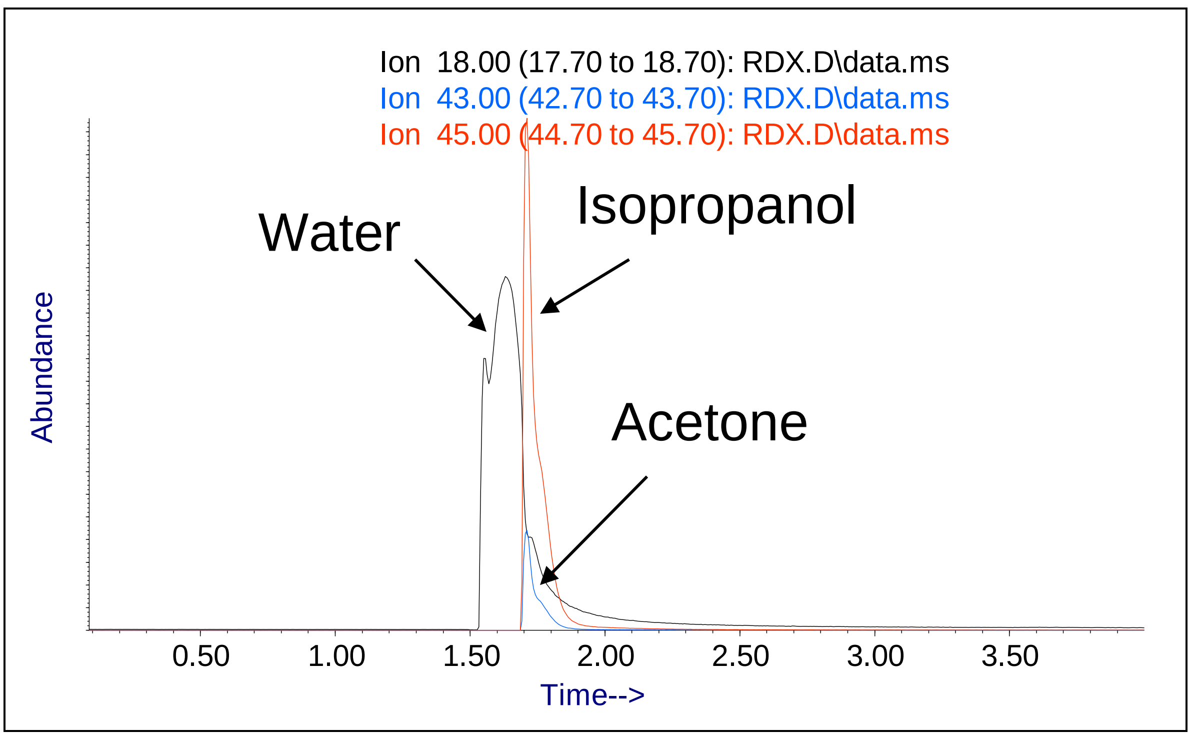

Figure A-8. Selected ion chromatograms resulting from D-GC-MS analysis of RDX.

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